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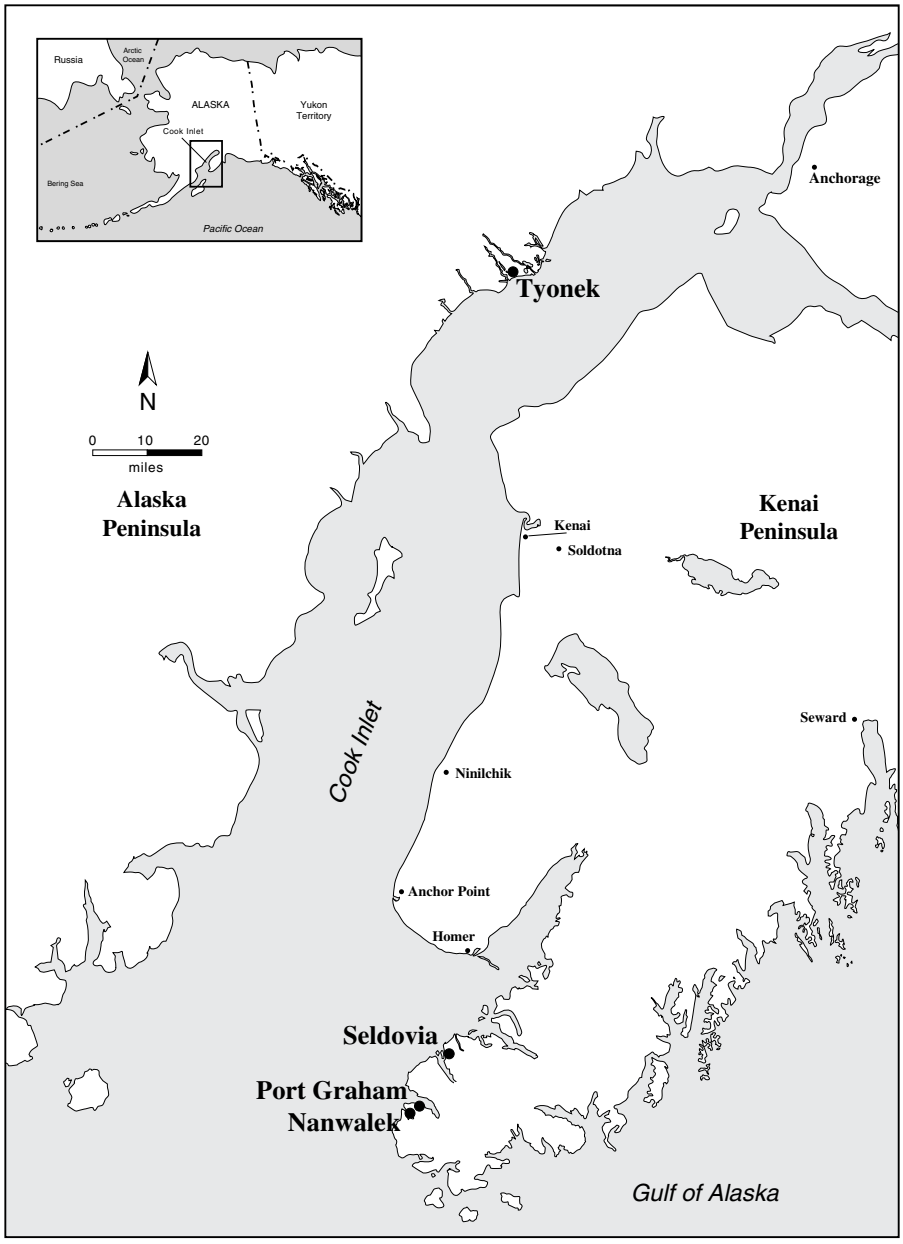
Appendix F

Study plan for conducting field sampling & and chemical analysis for Cook Inlet contaminant study



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Study Plan
for Conducting
Field Sampling and Chemical Analysis
for the
Cook Inlet Contaminant Study

October 1997

Prepared by

United States Environmental Protection Agency
Office of Water
Office of Science and Technology

TABLE OF CONTENTS

Acknowledgments

This report was prepared by the U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology. The EPA Study Manager for this study is Jeffrey Bigler, who is responsible for overall project coordination as well as technical direction. EPA is supported in the development of this document by DynCorp (EPA Contract No. 68-C3-0337) and Arthur D., Little (EPA IAG No. DW14937850-01-0). Lynn Riddick is Project Manager for DynCorp, and John Brown is Project Manager for Arthur D. Little. EPA personnel who have contributed to the development of this study include Marvin Rubin, Chief, Energy Branch, Engineering and Analysis Division; William Telliard, Chief, Analytical Methods Staff, Engineering and Analysis Division; Janet Goodwin, Project Manager for Chemical Analysis, Engineering and Analysis Division; Laurie Mann, Office of Water, EPA Region 10; and Rick Albright, Director, EPA's Alaska Operations Office.

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1.0 Introduction and Scope

The United States Environmental Protection Agency (EPA) is conducting a study of chemical contaminants in the Cook Inlet area of Alaska. The Cook Inlet Contaminant Study is being conducted by the EPA Office of Science and Technology (OST) as part of its effort to assess health risks to Native American tribes living in coastal Alaska. Specifically, the study will provide the information necessary to characterize health risks associated with exposure to contaminants detected in fish and wildlife harvested from Cook Inlet by members of four native Alaskan subsistence villages who are dependent on marine resources as a source of dietary food items. This human health risk assessment is part of a larger OST effort to characterize human health risks from pollutants associated with Offshore and Coastal Oil and Gas Industry practices.

In preparing to meet this commitment, the EPA Office of Water (OW) has contacted and held meetings with representatives from many State, Federal, Tribal and private organizations for the purpose of soliciting assistance and input in designing the study. These organizations include, among others, the Alaska Department of Fish and Game (ADF&G), the Alaska Department of Health and Social Services, the National Marine Fisheries Service (NMFS), the Minerals Management Service, the Alaskan Village Councils and Chiefs for Tyonek, Seldovia, Port Graham and Nanwalek, and the Harbor Seal Commission.

The study design, sampling methods and protocols, and analytical methods and protocols used in conducting this study are based on EPA peer reviewed documents. Any deviations from the guidance contained in these peer reviewed documents will be described within the final report.

The field sample collection phase of the study was scheduled to begin during the week last week of May 1997, and was to be completed by mid summer. The sample collection includes the assistance of the harvesters from all four villages, biologists from the Alaska Department of Fish and Game and EPA contract personnel. Items sampled from Cook Inlet include anadromous and resident fishes, marine invertebrates, marine plants and marine mammals. The appropriate permits for collection of specimens were to be secured by EPA from ADF&G, NMFS and the International Pacific Halibut Commission. The samples were to be collected in cooperation with the four villages of Tyonek, Seldovia, Port Graham and Nanwalek.

The laboratory analysis of all samples will be completed in early 1998, with the risk analysis phase of the study expected to be completed during late spring 1998. The risk analysis phase of the study will involve the formation of an advisory committee, and if necessary, a risk communication advisory committee will also be formed. We anticipate that a final report describing the results of this study will be released in late spring 1998.

This document summarizes the study plan for conducting the Cook Inlet Study. Information contained in this plan has largely been extracted from The Quality Assurance Project Plans for Field Sampling[1] and Sample Analysis[2], which are attached.

2.0 Project Organization

The Office of Science and Technology is responsible for overall management of the study; day-to-day responsibility for managing various aspects of the study have been delegated to the Standards and Applied Science Division (SASD) and the Engineering and Analysis Division (EAD) within OST. SASD is responsible for managing all sample collection and data analysis activities associated with this study; EAD is responsible for managing all laboratory analysis and data verification (data review) activities. Both EAD and SASD are responsible for day-to-day interaction with contractors and with other federal, state, and local authorities involved in the project

Several federal, state, local, and contractor organizations are participating in this project. These include EPA, the National Marine Fisheries Service (NMFS), the Alaska Department of Fish and Game (AF&G), the Harbor Seal Commission (HSC), DynCorp Information & Engineering Technology (DynCorp), Arthur D. Little Inc. (ADL), and several Village Coordinators from the villages of Tyonek, Seldovia, Port Graham, and Nanwalek, Alaska..

The following describes roles and responsibilities of EPA study management personnel and staff at the Sample Control Center (SCC). Additionally, the capacity to which contractors will support this study is identified. Figure 1 illustrates the relationships and lines of communications between each of these organization.

EPA Staff

OST Director - The OST Director, Tudor Davies, is responsible for providing financial and staff resources necessary to meet study objectives and implement study requirements.

OST QA Manager - The OST QA Manager, William Telliard, is responsible for assisting the EPA Study Manager and Project Managers with the development and implementation of QAPPs[1,2] for this study. The QA Manager also is responsible for ensuring that all QA procedures are followed, reporting any deviations from the QAPPs to the Project Managers, and assisting the Project Managers in implementing corrective actions necessary to resolve these deviations. The QA Manager reports directly to the OST Director.

OST Study Manager - The OST Study Manager, Jeffrey Bigler, is responsible for providing overall direction concerning the study to the EPA Project Managers shown in Figure 1. The OST Study Manager also is responsible for:

- Developing and implementing an overall Study Plan applicable to all phases of the Cook Inlet Contaminant Study
- Communicating study objectives to the EPA Project Managers shown in Figure 1
- Reviewing and approving all major work products associated with the study
- Participating in meetings with the EPA Project Managers, other EPA staff, and

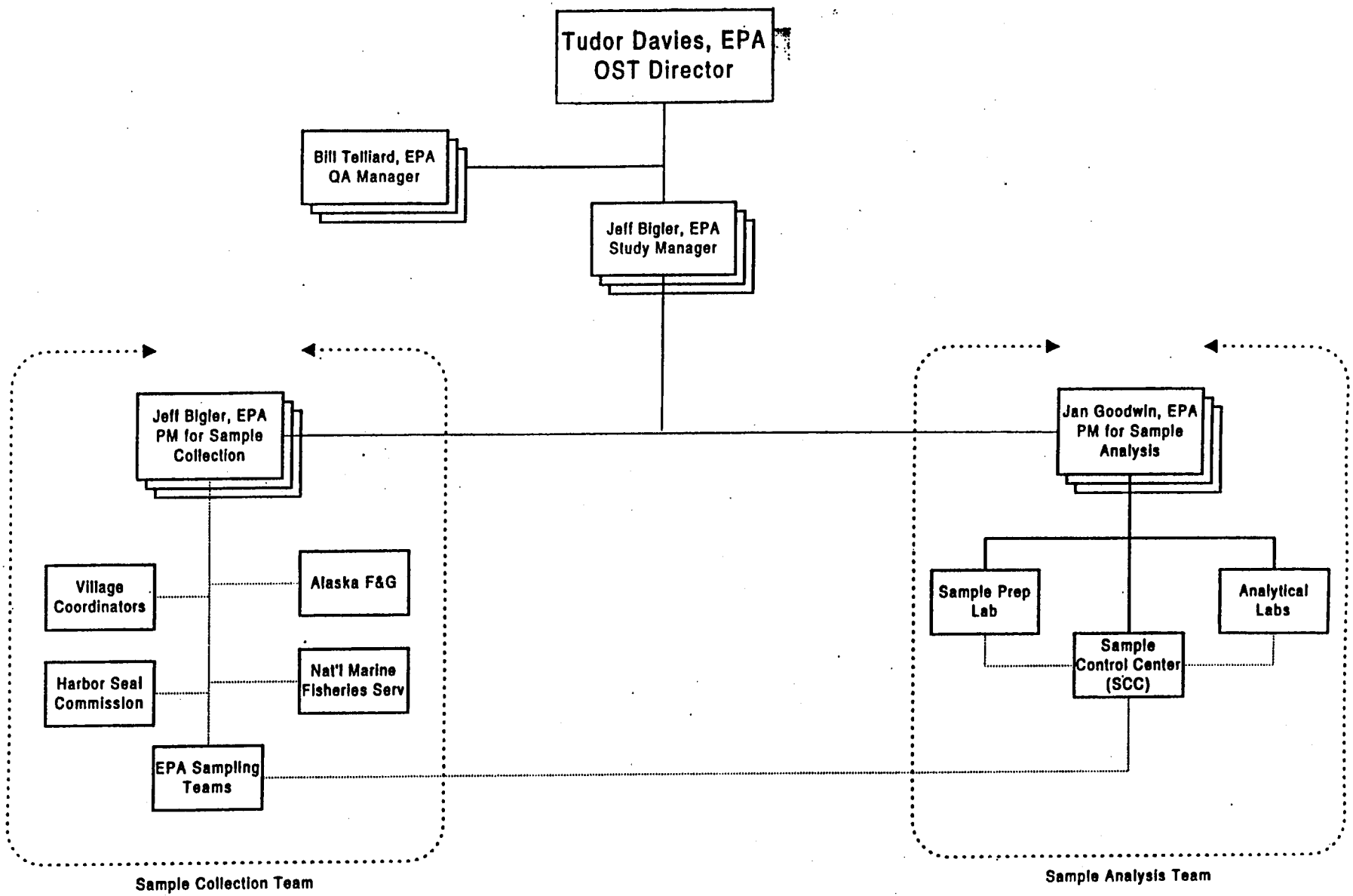


Figure 1 - Project Organization

- staff from other organizations and contractors concerning the study
- Working with the OST QA Manager to identify corrective actions necessary to ensure that study objectives are met.

Project Manager for Sample Analysis - The OST Project Manager for Sample Analysis, Jan Goodwin, is responsible for:

- Developing and implementing the Sample Analysis QAPP[2]
- Daily oversight of EPA and contractor staff involved in activities related to analysis of samples collected in this study
- Communicating project objectives to all EPA and contractor staff involved in the analysis of samples collected in this study
- Reviewing and approving major deliverables related to the analysis of samples collected in this study
- Participating in meetings with the OST Project Manager for Sample Collection, the OST QA Manager, the OST Study Manager and the OST Director concerning study objectives, schedules, and concerns
- Providing technical assistance concerning sample analysis and data evaluation to the OST Project Manager for Sample Collection

Project Manager for Sample Collection - The OST Project Manager for Sample Collection, Jeff Bigler, is responsible for:

- Developing and implementing the field sampling QAPP[1]
- Daily oversight of EPA and contractor staff involved in activities related to sample collection in this study
- Communicating project objectives to all EPA and contractor staff involved in the sample collection in this study
- Reviewing and approving major deliverables related to the sample collection in this study
- Participating in meetings with the OST QA Manager, the OST Project Manager for sample analysis and the OST Director concerning study objectives, schedules, and concerns
- Providing technical assistance concerning sample collection and data evaluation to the OST Project Manager for Sample Analysis.

Sample Control Center Staff (SCC) - The Sample Control Center (staffed by DynCorp) will be responsible for facilitating effective communication among all of the parties involved in the shipment and analysis of samples under this study. SCC also will be responsible documenting all sample shipments, problems that arise, and resolutions to those problems. Finally, SCC will be responsible for reviewing laboratory data to ensure that the measurement quality objectives (MQOs) are met, working with the laboratories and EPA to correct QC failures, where possible, and for documenting the extent to which data submissions meet study MQOs.

Contractor Support - In addition to the SCC activities, EPA obtains contract services for assisting in the field collection of samples. EPA also contracts laboratory services for the preparation and analysis of samples through its "megalab" contracts . At least two contract laboratories are involved in the study. One contract laboratory is responsible for all activities related to the homogenization, compositing, and/or aliquotting of samples. It is possible that this laboratory also will be issued a delivery order to perform some or all of the subsequent sample analyses. If necessary to maximize expertise and minimize costs, EPA also may issue delivery orders to one or more additional contract laboratories. Contractor support is also obtained for developing a final report which includes all phases of the study, including risk analysis.

3.0 Project Description

Under this project, marine plant, marine mammal, marine invertebrate, and fish samples will be collected from areas near four villages around Cook Inlet, Alaska: Tyonek, Seldovia, Port Graham, and Nanwalek. EPA will coordinate with the four Alaskan Villages to identify locations where samples of specific dietary food items are routinely harvested by the Village members. EPA and the designated Village Coordinators will coordinate the collection of whole samples of finfish, marine invertebrates, marine mammal organs, and marine plants from these identified areas during the summer of 1997. EPA personnel and/or EPA contractor support will provide direct oversite of the collection of all samples. All field collection protocols and methods will be consistent with recommendations included EPA document titled Guidance For Assessing Chemical Contaminant Data For Use in Fish Advisories Volume I: Fish Sampling and Analysis, Second Edition[3], or as approved by the EPA Study Manager.

Following collection, the samples will be frozen and shipped to a contract laboratory (the "Sample Prep Lab") for homogenization, compositing, and/or aliquotting. This laboratory must have the capability to store, homogenize, composite, and otherwise handle biological and aqueous samples for low level organic and inorganic analysis without contaminating the samples. To verify that these procedures are contaminant-free, the Sample Prep Lab will prepare and analyze equipment blanks with each batch of field samples prepared. These blanks will be analyzed for each of the target analytes listed in Table 1. If the Sample Prep Lab does not have the analytical capability to analyze for all of these target pollutants, it will ship the equipment blanks to a qualified laboratory, operating under an approved EPA contract or subcontract, for analysis of the required pollutants. Data demonstrating the absence of contamination will be sent to SCC for verification.

Upon completion of these activities, the Sample Prep Lab will ship frozen aliquots of the individual or composited samples to other contract or subcontract laboratories for analysis. Analytical laboratories will use the methods described in the Sample Analysis QAPP and specified in delivery orders issued under existing EPA contracts to prepare and analyze the samples.

Following sample shipment, SCC:

- maintains routine communication with the analytical laboratories to identify any significant questions, problems, or delays that arise during the course of sample analysis;
- reviews the sample analysis to ensure that the procedures followed and the results obtained are consistent with those specified by EPA;
- works with the laboratory to correct any identified problems prior to submitting the final, reviewed data packages to EPA;
- evaluates overall data quality against the MQOs and will prepare a written assessment of its findings;
- includes the written assessment with the final, reviewed data packages.

Upon receipt of the reviewed data packages and SCC data review summaries, EPA will make a final data usability determination of each data point for its coastal Alaska human health risk assessment.

Details regarding procedures and quality control requirements needed for the activities summarized above are documented in the Sample Analysis QAPP.

Table 1
List of Target Pollutants in Cook Inlet Contaminant Study

Organohalide pesticides
Dioxins/furans
Toxic PCBs
Hydrocarbons, Phenols, Polycyclic Aromatic Hydrocarbons and PAH Metabolites
Total mercury
Methyl mercury
Total arsenic
Inorganic arsenic
Arsenic (III)
Arsenic (V)
Monomethylarsonic acid (MMA)
Dimethylarsonic acid (DMA)
Chromium
Selenium
Cadmium
Lead

4.0 Quality Objectives and Criteria

The following quality objectives and criteria are applicable to the sampling and analysis activities associated with the Cook Inlet Contaminant Study.

Sample Collection

This section primarily focuses on the sample collection effort. Discussion of conventional data quality measures within the context of the overall study are also included.

The overall quality objectives of the field collection effort are:

- Collect samples representative of subsistence foods
- Collect representative samples of the identified foods
- Minimize contamination of sampling during collection, handling, and shipment

The Field Sampling QAPP and sampling design described in Section 5.0 designed to address these specific objectives are summarized below:

To ensure that species representative of subsistence food are collected, EPA worked with the Village Leaders and Village Coordinators to properly identify target species and sample types.

To ensure that representative samples of the identified foods are collected, the sampling effort will be conducted during optimal tide conditions during a peak harvest period.

To minimize contamination of samples during collection, specific procedures will be followed as described in EPA guidance[3] and QAPP[1]. These include such requirements as the use of gloves for sample handling, clean glass jars, clean foil and polyethylene bags for sample packaging, and adherence to operating procedures for each collection effort.

Special Training Requirements or Certifications

The Study Manager is responsible for ensuring that all Field Sampling Staff are experienced in environmental activities involving the collection of biota, and that they are also be familiar with operation of fishing equipment and small boats.

The Field Sampling Task Leaders are responsible for ensuring the village gatherers are properly trained for collection of the different samples.

Documentation and Records

Field Records - Whenever a sample is collected, a detailed description of the sample will be

recorded on a Field Record Form. This record will document the sample type, location, and other sample details.

The actual samples are assigned tracking numbers which are also recorded on the form to identify the sample to the record. The samples are identified by use of sample tags with sample numbers, sampling locations, and date and time of collection.

Field Notes - Field notebooks will provide the means of recording field data collection activities. As such, entries will be described in as much detail as possible so that a particular event or situation could be recreated without reliance on memory.

Field notebooks will be bound survey books or notebooks and will be assigned to field sampling staff.. The title page of each notebook will contain the following:

- person to whom the notebook is assigned
- notebook number
- project name
- project task start and end dates

Entries into the notebook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of all sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered.

The longitude and latitude of the sampling location will be identified using a current NOAA charts which are accurate to approximately 0.1 nautical miles.

The equipment used to collect samples will be noted, as well as other pertinent sampling details including the time of sampling, sample description, depth at which the sample was collected, volume and number of sample containers.

Final Report Records - The final report will include narrative description of the field activities. Copies of field records will also be included in the report. Also included will be a summarized listing of samples identifications, sample types, and sampling locations.

Record Retention and Final Disposition - Original field records and field notebooks are to be maintained by the Study Manager at the completion of each individual task. Copies of these records will be maintained by the originator for reference should problems occur that require resolution.

Copies of all shipping logs, airbills, and other related documentation will maintained Study Manager.

Specification for retention of field samples by the receiving location are outside the scope of this document. While in storage, it is recommended that unused samples be stored with the original

labeling materials.

Sample Analysis

This section focuses on quality objectives related to measurement aspects, and are referred to as measurement quality objectives (MQOs).

MQOs for this project are described in terms of the following criteria: precision, accuracy, sensitivity, representativeness and completeness. Specific objectives for these criteria are provided below.

Precision - Precision is defined as the relative uncertainty about a given measurement and can be evaluated by comparing replicate sample results. In this study, overall analytical precision (i.e., precision associated with the sample homogenization, compositing, aliquotting, shipping, and analysis processes) will be assessed by evaluating results from duplicate sample aliquots prepared by the Sample Prep Lab and sent to each laboratory for analysis of the target pollutants.

The design of this study includes a large number of other QC samples that provide information about the precision associated with various components of the analytical process. Details regarding these process-specific QC samples and specific MQOs for each are given in the section titled QC Requirements.

Accuracy - Accuracy is defined as the degree of agreement between an observed (e.g., measured) value and an accepted reference, or "true" value. In this study, overall accuracy of the analytical process will be measured by preparing and analyzing spiked field samples. Depending on the method used for analysis of individual pollutants targeted in this study, the spiked field samples may take the form of (1) matrix spike samples, which are field samples spiked with the analytes of interest, (2) field samples spiked with isotopically labeled compounds, (3) field compounds spiked with surrogate compounds that are expected to behave in a manner similar to the target analytes but that are not expected to be present in the field samples collected, or (4) some combination of items 1 and 3.

The measurement quality objective for overall analytical accuracy in this study is for 90% of the spiked field sample results to fall within the applicable acceptance criteria listed below in Table 2.

Table 2: Measurement Quality Objectives for Accuracy*			
Analyte	Measure	Frequency	Method
Organohalide pesticides	Matrix spike and matrix spike duplicate (MS/MSD) samples	Spikes of all target analytes in 1 pair of samples per 10 samples of each matrix type	Modified Method 1656
	Surrogate compound spikes	At least one surrogate compounds spiked into every sample	
Dioxins/furans	Labeled compound spikes	Spikes of all target compounds in each sample	Method 1613, Revision B
Co-planar PCBs	Labeled compound spikes	Spikes of all target compounds in each sample	Method 1668
Hydrocarbons, Phenols, PAH/ PAHMetabolites	Labeled compound spikes	Spikes of all target compounds in each sample	GC/Selective detector
Total mercury	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1631
Methylmercury	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Research method
Total arsenic	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1638
Arsenic (III)	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Quartz furnace AA
Arsenic (V)	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Quartz furnace AA
MMA	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Quartz furnace AA
DMA	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Quartz furnace AA
Inorganic arsenic	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Quartz furnace AA
Total chromium	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1638
Seleneium	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1638
Cadmium	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1638
Lead	MS/MSD samples	1 pair of samples per 10 samples of each matrix type	Modified Method 1638

* Overall MQO for accuracy in study is for 90% of accuracy measurements to fall within the individual measurement MQOs listed in the table.

The design of this study includes a large number of additional QC samples that provide information about the accuracy associated with various components of the analytical process. Details regarding these process-specific QC samples and specific MQOs for each are given in the QAPP.

Sensitivity - Analytical sensitivity is defined as the minimum concentration of an analyte above which a data user can be confident that the analyte was reliably detected and quantified. For this study, the method detection limit (MDL) and the minimum level (ML) will be used to define the sensitivity of each measurement process.

The MDL is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.

The ML is defined as the lowest concentration at which the entire analytical system must give a recognizable signal and acceptable calibration point for an analyte. It is equivalent to the concentration of the lowest calibration standard analyzed by a specific analytical procedure, assuming that all the method-specified sample weights, volumes, and processing steps have been employed.

It is not feasible to specify numeric objectives concerning the sensitivity of most pollutants targeted in this study because published methods are not specific for analysis of these pollutants in marine plant or tissue matrices. General DQOs have been developed based on risk assessment needs and existing measurement capabilities and are discussed in the Sample Analysis QAPP.

Completeness - Completeness is defined in terms of the percentage of data that are collected and deemed to be acceptable for use in this study. Three measures of completeness can be defined, as follows:

- Sampling Completeness, which is the number of valid samples collected relative to the number of samples planned for collection;
- Analytical Completeness, which is the number of valid sample measurements relative to the number of valid samples collected; and
- Overall Completeness, which is the number of valid sample measurements relative to the number of samples planned for collection.

Completeness goals are presented in Table 3.

Table 3
Measurement Quality Objectives for Completeness

MQO	Measure	Analyte	Acceptance Criteria
Sampling completeness	Number of valid samples collected relative to the number of samples planned for collection	Not Applicable	90% (see reference 1)
Analytical completeness	Number of valid sample measurements relative to the number of valid samples collected	All	90%
Overall completeness	Number of valid sample measurements relative to the number of samples planned for collection	All	81% (determined by multiplying sampling and analytical completeness goals).

Representativeness - Representativeness is an indication of (1) the degree to which a sample from a given site is typical of that site or area (e.g., a coastal harbor) and the matrix from which it was taken (e.g., a tissue matrix), and (2) the degree to which the sample accounts for analyte heterogeneity in the matrix.

An in-depth study of sample homogeneity and the effectiveness of the compositing procedure will be performed with three sets of flounder samples collected from Port Graham Village. Flounder collected in these sets will be prepared as individuals and as composites, and the composite samples will be subsampled and replicated for subsequent analysis by the analytical laboratories. This representativeness study, which is illustrated in Figure 2, will be performed as specified in the Field Sampling QAPP.

Because resource constraints make it impractical to perform such an in-depth representativeness study in all Cook Inlet matrices sampled, the Sample Prep Lab will prepare duplicate samples at a frequency of 5% (1 per 20) per matrix per method/analyte as specified in the Field Sampling QAPP. The measurement quality objective for these Sample Prep Lab duplicates is± 30%.

5.0 Sampling Design and Sampling Methods

Approach

The sampling approach is designed to collect samples of marine organisms representative of the historic and routine harvest and consumption by the four Native Alaskan tribes. The types of samples were selected because they are commonly consumed in the area and are of high commercial, recreational, and subsistence fishing value based on interviews with Village members. The number of each type of sample and the compositing scheme is based EPA guidance[3] and is specified in the Field Sampling QAPP.

For practical reasons, the sampling design also considered species that are relatively abundant, easy to capture or collect, and large enough to provide sufficient tissue for chemical analysis. The samples collected are intended to be representative of those used by the Alaska Villages. As such, only legal harvestable sizes (as per subsistence guidelines) will be collected.

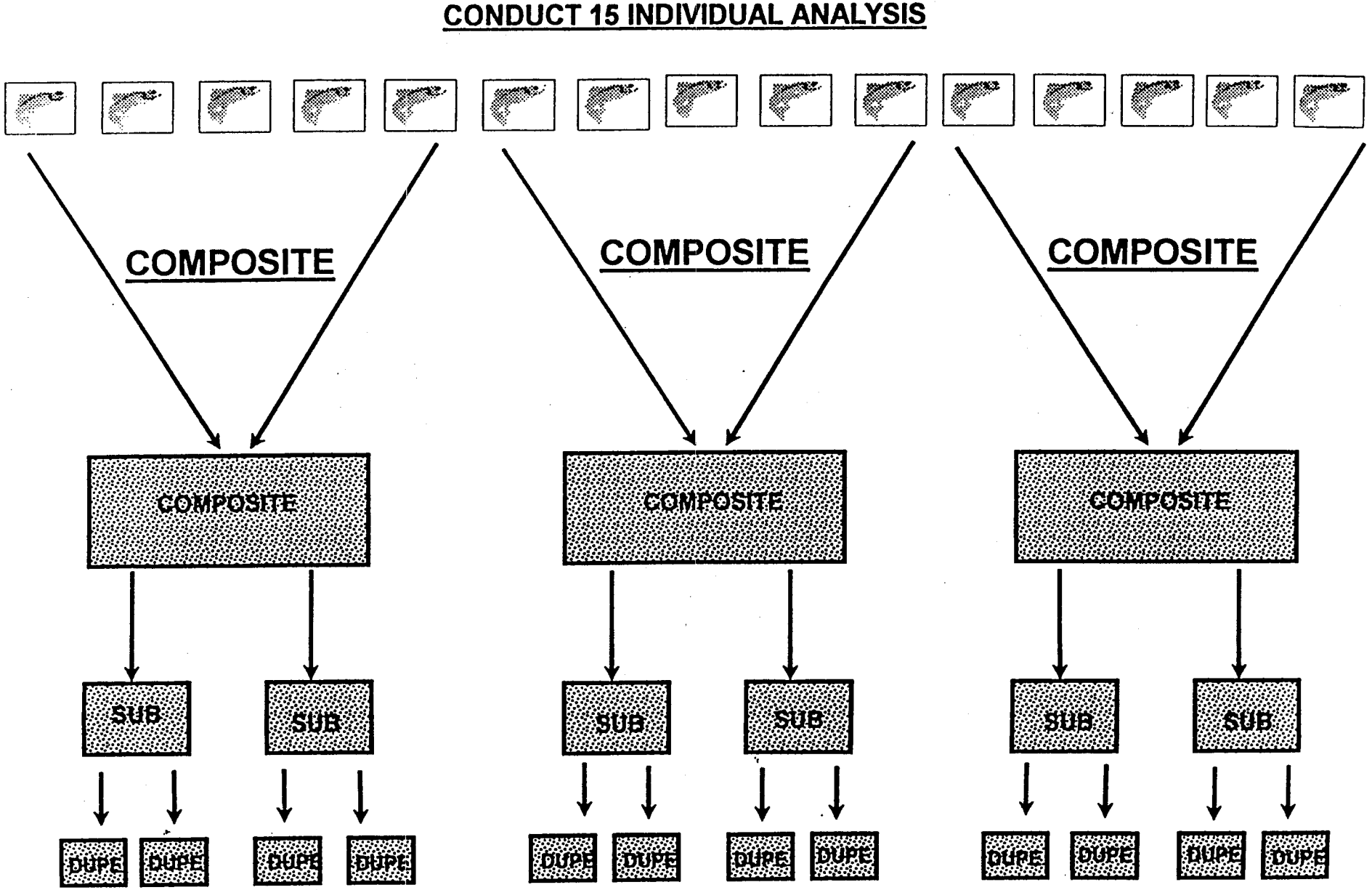
The collection volumes are designed considering the following compositing requirements:

- samples should be of similar size with the smallest length or weight no less than 75% of the largest
- samples are collected at the same time
- sufficient numbers are collected to provide adequate tissue for analysis

Sampling Plan

Each fish sample composite is comprised of five individual samples, with the possible exception of halibut where the number of individual samples may be reduced depending upon the

FLOUNDER ANALYSIS TO SUPPORT COMPOSITE TESTING



TOTAL NUMBER OF SAMPLES FOR ANALYSIS = 27 (15 INDIVIDUALS + 12 DUPES)

size of the available catch. Mammal samples will be comprised of one individual. Octopus samples will be comprised of individual organisms; not a composite. Plant and invertebrate composites will be comprised of a **minimum** of five individuals, but enough individuals to total approximately 300 grams of tissue sample. All sampling sites will be located within designated subsistence harvest areas

Sample Locations and Numbers

The following details the samples that will be collected in each area:

Tyonek Village
6 Whole Fish Composite Samples comprised of:
1 Chinook Salmon x 3 composites
1 Sockeye Salmon x 3 composites

6 Marine Mammal Organ Samples comprised of:
1 Beluga x 3 organs
1 Seal x 3 organs

Seldovia Village
7 Whole Fish Composite Samples comprised of:
1 Red Salmon x 3 composites
1 Chinook Salmon x 1 composites
2 resident species (e.g., Bullhead, Halibut) x 3 composites

15 Whole Invertebrate Composite Samples comprised of:
1 Clam species (e.g., Butter Clam) x 3 composites
1 chiton species x 3 composites
1 mussel species x 3 composites
1 snail species x 3 composites
1 clam species from Ninilchik area (e.g., Razor Clam) x 3 composites

Port Graham Village
11 Whole Fish Composite Samples comprised of:
1 Chum Salmon x 3 composites
1 Chinook Salmon x 2 composites
2 resident species (e.g., Flounder, Seabass) x 3 composites

12 Whole Invertebrate Composite Samples comprised of:
1 clam species (e.g., Butter Clam) x 3 composites
1 chiton species x 3 composites
1 octopus species x 3 individuals
1 snail species x 3 composites

6 Marine Plant Composite Samples comprised of:
1 kelp species x 3 composites
1 Goosetoungue Plant x 3 composites
(only edible portion of the plant samples are collected)

Nanwalek Village
9 Whole Fish Composite Samples comprised of:
1 Sockeye Salmon x 3 composites
2 resident species (e.g., Cod, Halibut) x 3 composites

12 Whole Invertebrate Composite Samples comprised of:
1 Clam species (e.g., Butter Clam) x 3 composites
1 chiton species x 3 composites
1 octopus species x 3 individuals
1 snail species x 3 composites

3 Marine Mammal Organ Samples comprised of:
1 Harbor Seal x 3 organs

6 Marine Plant Composite Samples comprised of:
1 kelp species x 3 composites
1 Goosetongue Plant x 3 composites
(only edible portion of the plant samples are collected)

Sampling Methods Requirements

This section describes the procedures for collecting samples.

Sample Integrity

A critical requirement of the project is the maintenance of sample integrity from the time of collection to the shipment and arrival at the final destination. Sample integrity is preserved by preventing the loss of contaminants that might be present in the sample and the prevention of the introduction of contaminants during handling.

The loss of contaminants can be prevented in the field by ensuring that the sample collected is intact. Sample collection procedures are to performed with the intention of minimizing the laceration of fish skin and the breakage of invertebrate shells or carpaces. Once collected, integrity is maintained through careful and controlled sample handling and storage and preservation procedures.

Sources of extraneous contamination can include contamination from the sampling gear, oils and greases on ships, spilled fuel, skin contact, contact with soil or sand, exhaust, as well as a number of other unanticipated sources. All potential sources should be identified before the onset and during sample collection, and appropriate measures should be taken to minimize or eliminate

them. The following are some examples:

- Boats should be positioned so that engine exhaust does not fall on the deck area where samples are being collected
- Ice chests and other sample storage containers should be scrubbed clean with detergent and rinsed with distilled water prior to use
- Samples should not be placed directly on melting ice, but should be stored inside plastic bags first
- Proper gloves should be used when handling samples and gloves should be discarded after each use

Specific Sampling Protocols

In addition to the methods and protocols recommended in EPA guidance[3], the following generally describes the sample collection procedures for the fish. Procedures for the collection of plants, invertebrates and mammals are included in the Field Sampling QAPP.

Collection Procedure for Fish Samples

Purpose

To collect fish samples which are representative (i.e., the same species and size ranges) of those harvested for subsistence purposes in the Villages of Seldovia, Nanwalek, Port Graham, and Tyonek Alaska. King (Chinook) salmon, red (sockeye) salmon, chum salmon, halibut, bullhead (sculpin), flounder, seabass and cod are the target species for collection, and will be analyzed for a suite of organic and inorganic contaminants.

Procedure

1. Identify the area (station) for collection of each composite sample. One composite sample of fish will be collected from each of three areas (stations) where village gatherers normally collect subsistence fish for red salmon, chum salmon, bullhead, halibut, seabass, flounder and cod. For king salmon, three composite samples will be collected from Tyonek, two composites will be collected from Port Graham, and one composite will be collected from Seldovia. The species to be collected at each village are as follows:

Seldovia: king salmon, red salmon, halibut and bullhead
Nanwalek: red salmon, halibut and cod
Port Graham: king salmon, chum salmon, seabass and flounder
Tyonek: king salmon and red salmon

The station where each composite sample is collected should be separate area if possible i.e., section of beach, area of a bay, etc. However, this may not be possible for all species collected within each village, where individual fish comprising a composite may be collected from different areas, or the same area at different times. Information should be carefully documented to indicate the collection locations and times for each composite sample.

2. Fish samples will be collected by set net and angling. Efforts should be made to minimize the use of tools or implements which may come in contact with the fish during collection (i.e., gaffs

may be necessary to land some of the larger species). After the fish is landed or removed from a net, it will be killed (metal implements should not be used to kill fish, a clean wooden bat is recommended) and rinsed in ambient seawater at the collection site to remove sand particles and rock fragments.

3. For the king salmon and halibut, each fish will be placed in a heavy duty food grade polyethylene bag, and sealed with a cable tie. A completed sample ID label will be affixed to the cable tie and the bag will be placed inside another polyethylene bag and sealed with another cable tie. All other fish will be wrapped in aluminum foil (pre-rinsed with acetone and baked at 400 °C) before being "double bagged" and labeled as described above. For boat landed fish, extra care should be taken to keep the fish from contacting any sheen from the boat engine. At no time will the fish be allowed to contact the bilge water in the boat. Clean nitrile gloves will be worn at all time during the cleaning and handling of the fish samples.

4. Each composite fish sample must be collected as 5 individual fish at each station. As noted previously, the "station" may encompass a large area (i.e., a bay), and the fish from one station may be collected on different days. For the Tyonek salmon, it is expected that the fish will be collected from set nets in one day for each species. The stations for Tyonek salmon collection should located such that each of the three composite samples are obtained from different areas of the set net beach (i.e., north area, middle area, and south area).

5. Field blanks will be collected during the fish sampling to determine the potential contribution of target contaminants from the sampling containers (polyethylene bags and foil) and shipping. A field blank will be prepared by filling a sampling bag with ~6 liters of commercially available distilled (DI) water and wrapping the bag in the same manner as the fish samples (i.e., double bags with sample ID label). The field blank will be frozen as soon as possible, and packaged and shipped in the same manner as the fish samples. Five fish field blanks will be collected during the course of the study: 1 with DI water only (Tyonek king salmon sample poly bags), 2 with DI water only (4 mil poly bags) and 2 with DI water and ~ 3 ft of rinsed and baked aluminum foil (4 mil poly bags).

Storage

During the collection of the composite fish sample, the individual fish will be stored on ice (a maximum of 24 hours). Within 24 hours the samples should be stored frozen (-20°C) until shipment. The fish samples from Tyonek will be stored frozen at 10th and M Seafood (Anchorage, Alaska) until shipment.

Documentation

1. For fish samples in polyethylene bags the sample ID label will be completed in permanent ink and affixed to the cable tie of the inner bag, no tape will be used to seal the bags.

2. A field record form should be completed for each composite fish sample. Information on the location of the station, sample identification, date and time of collection, collector's name(s), species, number of individuals, length of each individual, and a diagram of the collection site should be included on the field record form. Any additional information, as well as a log of daily sampling activities, should be noted in the field collection notebook.

3. Photo documentation will be collected from at least one sampling site for each species of fish collected. At a minimum, the photo documentation will include a picture of the collection site (station), a picture of a representative species, and a picture of the collection process.

Shipping

The fish samples will be shipped frozen to the designated laboratory(s) for chemical analysis. The fish will be packed in coolers for shipment. Excess space in the cooler will be filled with plastic "bubble wrap" to reduce the shifting of the samples during shipment. The cooler will be packed with dry ice (~20-30 lbs per cooler) to maintain a temperature below -4°C until receipt at the laboratory. A chain-of-custody form providing a listing of the sample ID of each sample included in the cooler will be filled out, signed and included in a sealed plastic bag in each cooler. After the final packing of each cooler for shipment (i.e. adding dry ice) the cooler will be sealed with fiberglass reinforced strapping tape, and a custody seal will be affixed across the lid of the cooler.

All coolers containing fish samples from Seldovia, Port Graham and Nanwalek will shipped via Federal Express Next Day Air from Homer Alaska. Federal Express will be notified of the shipment by 9:00 AM on the day of shipment, and will guarantee next-day delivery to Chicago, IL or San Diego, CA, if the samples are ready for pick-up in Homer by 12:00 noon. The fish samples from Tyonek will be shipped by 10th and M Seafood via Federal Express. The Tyonek fish will be shipped frozen in wax coated "fish boxes". The fish samples (which are "double bagged" in the field) will be wrapped in foil, packed in dry ice, and the boxes filled with "bubble wrap" for insulation. Chain-of-custody forms will be included in each fish box and a custody seal will be taped over the opening of each box prior to shipment.

Quality Control Requirements

To monitor the potential contamination of samples from the sample containers, shipping containers, packing and the shipping process field blanks will be prepared in the field and shipped with the samples to the analytical laboratory. Procedures for ensuring the integrity of these activities are detailed in the Field Sampling QAPP.

6.0 Sample Handling and Custody Requirements

Sample handling procedures are described in the individual sampling protocols. All individual samples and sample composites will be transferred to the receiving laboratory under chain-of-custody. The chain-of-custody form acts as a record of sample shipment and a catalog of the contents of each shipment. Other general provisions for sample handling and custody are described within the Field Sampling QAPP. Once samples have reached the laboratories for preparation, formal chain of custody procedures will not be used. Instead, the laboratories will follow the general sample handling and documentation procedures that are routinely used by EPA. These standard EPA procedures are summarized in the Sample Analysis QAPP.

7.0 Analytical Methods Requirements

Samples collected under this study will be analyzed for organochlorine pesticides,

dioxins/furans, co-planar PCBs, hydrocarbons, phenols, polycyclic aromatic hydrocarbons (PAHs) and PAH metabolites, total mercury, methyl mercury, total arsenic, arsenic (III), arsenic (V), monomethylarsonic acid, dimethylarsonic acid, inorganic arsenic,, total chromium, selenium, cadmium, and lead. These pollutants are listed in Table 1.

Published EPA methods do not exist specifically for analyzing all the target pollutants in the marine plant and tissue matrices to be sampled in this study. Therefore, the following approach will be taken to identify appropriate methodology:

- If feasible, existing EPA methods will be modified to accommodate the matrices or pollutants of interest. In such cases, modifications will be thoroughly documented by EPA in an Analytical Requirements Summary issued as part of a delivery order under a megalab contract.
- If existing EPA methods cannot be identified, environmental analysis methods published by other organizations, such as the American Society of Test Materials (ASTM) and the Food and Drug Administration (FDA), will be evaluated. Where appropriate, these methods will be specified for use as written or with modifications. These specifications will be documented by EPA in an Analytical Requirements Summary issued as part of a delivery order under a megalab contract.
- If published methods are not available at all, methodologies described in public literature will be evaluated. Where appropriate, these techniques will be documented by EPA in an Analytical Requirements Summary issued as part of a delivery order under a megalab contract.
- If no methods have been published by EPA or other organizations, and if no clearly suitable methods can be identified in the public literature, EPA will specify general analytical needs, in terms of matrices, target pollutants, and required sensitivity, in an Analytical Requirements Summary issued as part of a delivery order under a megalab contract. EPA also will request that laboratories submitting bids to perform work under this delivery order also submit recommendations concerning appropriate methodology, the rationale for selecting this methodology, and sensitivity and performance criteria that might be expected when using this methodology on the matrices being sampled in this study. EPA will select a technique after evaluating the recommendations selected.

Information summarizing the methodologies that EPA expects to use in the Cook Inlet Contaminant Study are provided in the Sample Analysis QAPP. It should be noted that alternate techniques may be considered and implemented based on further discussions with experts in the field of environmental chemistry. In such cases, these alternate techniques will be documented in the Analytical Requirements Summaries issued by EPA.

8.0 Quality Control Requirements

All field sample analyses performed in this study will be performed in conjunction with the standard QC elements described in the EPA 600 and 1600 series methods. In developing these methods, EPA sought scientific and technical adv ice from many sources, including EPA's Science Advisory Board, scientists at EPA's environmental research laboratories, scientists in

industry and academia, and scientists, managers and legal staff at EPA Headquarters. The result of discussions held among these groups was the standardized QA/QC approach that is an integral part of the 600- and 1600- series methods. Over the years, these QA/QC requirements have been refined to reflect improvements in environmental science and policy. The most current versions of the 600- and 1600- series QA/QC are included in the Sample Analysis

12.0 Data Management

Field Data Management

Samples are tracked using the field records, field notebooks, and chain-of-custody forms. Because the sampling effort is a cooperative one involving a number of local sampling teams, the diligence of the field staff in completion of the proper records is essential.

Upon completion of the sampling effort, a database of samples and sampling locations will be constructed by EPA from the project documentation. This data summary will be included in the final project report.

To track shipments of sample coolers, only shipping services which include tracking of packages (e.g., FedEx) will be used.

Laboratory Data Management

Laboratory data management procedures can be summarized as follows. Each laboratory participating in this study will be required to maintain all records and documentation associated with the preparation and analysis of Cook Inlet Contaminant Study samples for a minimum period of three years. To facilitate data tracking, each laboratory will be required to utilize EPA-assigned episode and sample numbers when reporting results. All results of sample analyses, labeled and native standards, surrogate compounds, spike compounds, and blanks must be reported on hardcopy and on magnetic media. All reports and documentation required, including chromatograms and mass spectra, must be sequentially paginated and clearly labeled with the laboratory name, EPA Contract number, episode number, and associated EPA sample numbers. Any diskettes, or other electronic media submitted must be similarly labeled. Unless otherwise approved, the laboratories will use the hardcopy reporting forms and automated data reporting formats specified in the megalab contract when reporting data. Finally, each of the laboratories will adhere to a comprehensive data management plan that is consistent with the principles set forth in *Good Automated Laboratory Practices*, EPA Office of Administration and Resources Management, Draft, December 28, 1990. This data management plan must be in place and in use at all times during performance of the megalab contracts.

SCC Data Management

Data management procedures employed by SCC will include the use of 1) an automated

scheduling and tracking system to effectively manage data review and database development activities, 2) standardized data review guidelines to promote consistency in data quality audits across reviewers and over time, 3) a multi-stage data review process designed to maximize the amount of useable data generated in each study, and 4) a standardized data development process to facilitates rapid development of an analytical database with at least 99.9% accuracy.

The automated sample scheduling and tracking system will facilitate the development of up - to-date information concerning work in progress, projected delivery dates, and notice of any problems encountered with laboratory analyses or data turnaround times. To ensure that this information is as complete and accurate as possible, entries will be made into the tracking system at each stage of the sample-to-data sequence.

Detailed SCC data management procedures are provided in the Sample Analysis QAPP.

14.0 Assessments and Response Actions

Several types of assessment activities and corresponding response actions have been identified to ensure that data gathering activities in the Cook Inlet Contaminant Study are conducted as prescribed and to ensure that the measurement quality objectives and the data quality objectives established by EPA are met. These activities follow.

Field Sampling

To allow for oversight, all sampling teams will include at least one EPA staff or contractor field biologist to supervise all sample collection activities. Additionally, each field team supervisor will communicate the status of the project to the EPA Study Manager on a daily basis. The EPA Study Manager will consult with the Analytical Project Officer and QA officer regarding any difficulties encountered during field collection activities.

Chemical Analysis

More intensive assessment activities and corresponding response actions related to the chemical analysis phase of the study have been incorporated and are summarized in Table 5 and discussed in greater within the Sample Analysis QAPP.

15.0 Reports to Management

Upon completion of the field activities, the project will be summarized in report form and will become part of the final study report. Following completion of each data quality audit and assessment, SCC chemists will prepare and submit written reports, in narrative format, that describe data quality limitations and SCC recommendations concerning data use. This also will become part of the final study report.

16.0 Data Review, Validation, and Verification

All field notes and COC records will be reviewed by the Study Manager and QA Manager for completeness and correctness. Any discrepancies in field records will be reconciled with the associated field sampling personnel.

A multi-stage data review process, as summarized earlier and detailed in the Sample Analysis QAPP, will be used to evaluate the quality of all data submitted in the Cook Inlet Contaminant Study. Acceptance criteria against which analytical data will be evaluated will include 1) DQOs and MQOs detailed in the Sample Analysis QAPP, 2) applicable QC acceptance criteria outlined in the methods,3) best professional judgement of chemists responsible for performing data quality assessments. The analytical data review process is described in the Sample Analysis QAPP.

The EPA Study Manager will utilize the SCC-generated data review narratives to make final data usability determinations for each set of sample results for performing risk assessment activities.

References

(1) Combined Workplan and Quality Assurance Project Plan for the Cook Inlet Contaminant Study, Prepared for EPA by Arthur D. Little, Inc., June 5, 1997.

(2) Quality Assurance Project Plan for Sample Homogenization, Compositing, and Analysis in the Cook Inlet Contaminant Study, Prepared for EPA by DynCorp Environmental, September 1997.

(3) *Guidance For Assessing Chemical Contaminant Data For Use in Fish Advisories Volume I: Fish Sampling and Analysis, Second Edition*, July 1997; EPA 823-97-B-009.